
IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No. : 10/667,878
Applicant(s) : Henry Drummond Boswell et al.
Filed : 09/22/2003
Title : Oxidative Treatment of Hair with Reduced Hair
Damage
TC/AU. : 1615
Examiner : Eisa B. Elhilo
Conf. No. : 1156
Docket No. : CM2517M2C
Customer No. : 27752

DECLARATION UNDER 37 CFR §1.132

Commissioner for Patents
P. O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

I, Jennifer Mary Marsh, do hereby declare that I received a B.A. in Chemistry from Oxford University in Oxford, United Kingdom, and a Ph.D. in Inorganic Chemistry from Oxford University in Oxford, United Kingdom. I have been employed by The Procter & Gamble Company as a Principal Scientist from 1995 until present. I further declare that I have been involved in the development of hair colorant compositions for about seven (7) years.

I am a co-inventor of the above captioned patent application and am familiar with the subject matter contained therein. I am familiar with the Office Actions dated July 13, 2005, and the references cited therein. I personally performed the experiments described below which contrast the lightening and the hair damage resulting from treatment with compositions

of the present application and with oxidative hair color compositions described in U.S. Patent No. 6,004,355 to Dias et al. ("Dias"). The emulsion base used in the experiments described below was made by Colin Clarke, who also is employed by the Procter & Gamble Company.

EXPERIMENTAL METHODS

The following experiments were carried out to contrast the hair damage, as measured by the Goniophotometer Damage Assessing Protocol after a 5-Cycle Oxidative Hair Treatment Protocol With 10 Intermediate Washes, resulting in hair treated with compositions of the present application and with compositions comparable to Example A of Dias.

The lightening and hair damage results of ten products were contrasted for the present experiments. All the products were made for the present experiments using a standard emulsion base as the carrier for the bleaching actives, as outlined in the Appendix 1. This emulsion base was the same as that disclosed in the present application. The ten products can be described generally as follows where the levels are in the final mixed product as applied to the hair (which is mixed in a 1:1 ratio immediately before use).

Product 1: 1.32M hydrogen peroxide + 0.025% EDDS at pH 10

Product 2: 1.32M hydrogen peroxide + 0.05% EDDS at pH 10

Product 3: 1.32M hydrogen peroxide + 0.1% EDDS at pH 10

Product 4: 1.32M hydrogen peroxide + 0.2% EDDS at pH 10

Product 5: 1.32M hydrogen peroxide + 0.3% EDDS at pH 10

Product 6: 1.32M hydrogen peroxide + 1.0% EDDS at pH 10

Product 7: 1.32M hydrogen peroxide + 4.0% EDDS at pH 10

Product 8: 1.32M hydrogen peroxide + 0.05% EDTA at pH 10

Product 9: 1.32M hydrogen peroxide + 0.1% EDTA at pH 10

Product 10: 1.32M hydrogen peroxide + 1.0% EDTA at pH 10

Reasons for choice of product formulations:

- All the products contained a total of 1.32M oxidant. This is the total level of oxidant used in the 5-Cycle Oxidative Hair Treatment Protocol With 2 and 10 Intermediate Washes as described in the present application.

- All products were made with the same formulation described in the original application and at the same pH (pH 10).
- Product 6 contains EDTA at 0.05%, a standard level of chelant used in hair colourant formulations.
- Product 9 contains EDTA at 0.1%, as comparable to Example A of Dias.
- No dyes were used in the products as we were measuring the lightening performance of the products.
- The formulation with 4% EDDS was thickened with the addition of 1% of a xanthan gum polymer (Keltro HP from CP Kelco).

For the treatment of the hair, 1.5g 'virgin' switches were used, sourced from Hugo Royer International Ltd, 10 Lakeside Business Park, Swan Park, Sandhurst, Berkshire, GU47 9ND, United Kingdom. "Virgin hair" means hair that has never been treated chemically. For each product, 3 of these 1.5g switches were used.

2g of the product per 1g of hair to be treated was applied on the hair switches and massaged in thoroughly. The hair switches were then wrapped in a plastic film and put in an oven at 30°C. After 30 minutes, the hair switches were removed from the oven and from the wrapping film, and were rinsed for 1 minute in water. 0.1g of shampoo per 1g of hair was then added and milked for 30 seconds at a rate of at least 150 strokes a minute before rinsing for 30 seconds. The concentration of copper (Cu^{2+}) ions was kept at about 1 ppm (+/- 10%), the exact concentrations being measured by a standard analytic method. The rinsing water flow rate was 6 liters per minute. The same shampooing and rinsing process was repeated for a total of 10 times for the 10 wash protocol. This treatment cycle was repeated for five cycles total, and was the same as the one described as the "5-Cycle Oxidative Hair Treatment Protocol With 10 Intermediate Washes" in the present application.

Measurement of colour and lightening was performed on a Hunter Labscan Spectrophotometer. Details on this equipment are given in Appendix 2.

Fibre damage was measured for the 5 repeat cycles with 10 intermediate washes using a goniophotometer method and analysing the normalised shine from at least 100 single fibres.

Details on the equipment used and the test protocol are given in Appendix 3.

For the 5 repeat cycles with 10 washes we also checked the visual appearance of the fibres using a Scanning Electron Microscope and used a 4-point grading scale to assess the fibre damage. The protocol for this test is given in Appendix 4.

RESULTS

Normalized Shine Ratio as Measured by the Goniophotometer

Table 1 below shows the lightening values for the hair after 5 cycles of treatment expressed as dL (i.e., the increase in lightening after 5 cycles) and the normalised shine as measured by the goniophotometer. This normalised shine is compared to the normalised shine of the virgin starting substrate as a ratio of the shine of the test leg divided by the virgin shine. For each test leg at least 100 single fibres were measured.

Table 1.

	Product	Lightening (dL)	Normalised Shine	Ratio of Normalised Shine/ Virgin
	Virgin	-	12.89	
1	0.025% EDDS	24.4	12.20	0.95
2	0.05% EDDS	23.8	13.16	1.02
3	0.1% EDDS	23.7	13.04	1.01
4	0.2% EDDS	24.7	13.29	1.03
5	0.3% EDDS	23.9	12.74	0.99
6	1.0% EDDS	22.5	13.26	1.03
7	4.0% EDDS	21.3	12.70	0.99
8	0.05% EDTA	20.8	10.75	0.83

9	0.1% EDTA	25.2	10.99	0.85
10	1.0% EDTA*	22.8	8.08	0.70*

* Data previously generated as part of original work conducted in relation to present application (i.e., a different set of hair used wherein virgin normalised shine data = 11.54)

Scanning Electron Microscope Visual Assessment

Table 2 shows the results of the SEM grading (see Appendix 4 for details)

Table 2.

	Product	SEM Grading Scale Results				
		Low	Medium	High	Stripped	Damage Index
	Virgin	77	22	1	0	5.0
1	0.025% EDDS	16	71	6	7	24.8
2	0.05% EDDS	27	69	3	1	16.6
3	0.1% EDDS	72	21	7	0	8.4
4	0.2% EDDS	76	16	6	2	8.8
5	0.3% EDDS	59	33	8	0	11.4
6	1.0% EDDS	89	9	2	0	3.0
7	4.0% EDDS	73	19	5	3	9.8
8	0.05% EDTA	0	20	68	12	56.8
9	0.1% EDTA	0	5	81	14	63.6
10	1.0% EDTA*	1	19	38	42	68.6

* Data previously generated as part of original work conducted in relation to present application

CONCLUSION

It is the Examiner's opinion that U.S. Patent No. 6,004,355 to Dias et al. ("Dias") teaches hair dyeing compositions comprising the same ingredients in the same amounts as the compositions of the present application, and, therefore, the hair dyeing compositions of Dias would inherently possess the same physical properties as the compositions of the present application, such as a Normalized Shine Ratio as currently claimed. Based upon the results of the experiments described above, it is my conclusion that the compositions comprising EDTA as the chelant, which are comparable to the Example A composition of Dias, do not possess the same hair damage benefit as compositions comprising EDDS as the chelant, which are representative of the claimed compositions of the present application.

For example, in Table 1 it can be seen that Product 9, which comprises 0.1% EDTA, resulted in a Normalized Shine Ratio of 0.85. This value is less than the currently claimed Normalized Shine Ratio of at least about 0.95. Notably, even as the level of EDTA is increased, the resulting Normalized Shine Ratio does not increase. In contrast, Product 3, which comprises 0.1% EDDS, resulted in a Normalized Shine Ratio of 1.01. This value is greater than the claimed Normalized Shine Ratio of at least about 0.95. As the level of EDDS is varied, the resulting Normalized Shine Ratio remains above the claimed value of at least about 0.95.

Differences in resulting hair damage between the compositions comprising EDTA and the compositions comprising EDDS also are demonstrated by the SEM Grading results provided in Table 2. Clearly, the compositions comprising EDTA result in much more hair damage as compared to the compositions comprising EDDS at the same level.

Therefore, it cannot be assumed that the compositions of Dias inherently possess the same damage benefit properties as the claimed compositions of the present application.

This declaration is made with the knowledge that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed true, and further that willful false statements and the like are punishable by fine or

imprisonment or both, under 18 USC §1001 and may jeopardize the validity of the application or any patent issuing thereon.

13th Jan 2006

Date

Jennifer Marsh

Jennifer Mary Marsh

Declarant

APPENDIX 1FORMULATION OF PRODUCTSA. Formulation of the Emulsion Base Premix1. Protocol

- 1 Add cold water and commence agitation
- 2 Heat water to 82 C
- 3 Add EDTA, Na Benzoate, Cetearth-25
- 4 Add Stearyl/Cetyl Alcohol and Phenoxyethanol
- 5 Hold for 45 Mins at 80 C
- 6 Cool to 60 C whilst milling at 4000rpm using Turrax T50
- 7 At 60 C, stop milling and continue cooling to 50 C
- 8 Hold at 50 C for 1 hour
- 9 Check pH and allow to cool to 30 C
- 10 Pack off product

2. Ingredients

<u>Description</u>	<u>Wt %</u>
DI Water	82.6833
Cetyl Alcohol	6.2500
Stearyl Alcohol	6.2500
Cetearth 25	4.1667
Phenoxyethanol	0.3000
Sodium Benzoate	0.2500
Tetra Sodium EDTA	0.1000

B. Formulation of Final Tint Products1. Part A - Products 1-6 and Products 8-9Protocol:

- 1 Add water
- 2 Add ascorbic acid & sodium sulphite
- 3 Add chelant
- 5 Add ammonium hydroxide
- 6 pH adjust with acetic acid to required pH

7. Add solution to emulsion base slowly and mix thoroughly
8. Qs with DI water

Ingredients

a. Product 1

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	35.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Ammonium Hydroxide (30% active)	4.0000
EDDS chelating agent (30% active)	0.1700
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

b. Product 2

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	35.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Ammonium Hydroxide (30% active)	4.0000
EDDS chelating agent (30% active)	0.3400
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

c. Product 3

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	35.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Ammonium Hydroxide (30% active)	4.0000
EDDS chelating agent (30% active)	0.6700
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

d. Product 4

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	35.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Ammonium Hydroxide (30% active)	4.0000
EDDS chelating agent (30% active)	1.3400
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

e. Product 5

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	30.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Ammonium Hydroxide (30% active)	4.0000
EDDS chelating agent (30% active)	2.0100
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

f. Product 6

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	25.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Ammonium Hydroxide (30% active)	4.0000
EDDS chelating agent (30% active)	6.7000
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

g. Product 8

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	35.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Ammonium Hydroxide (30% active)	4.0000
EDTA chelating agent	0.1000
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

h. Product 9

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	35.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Ammonium Hydroxide (30% active)	4.0000
EDTA chelating agent	0.2000
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

i. Product 10

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	35.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Ammonium Hydroxide (30% active)	4.0000
EDTA chelating agent	1.0000
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

j. Product 11

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	35.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000

Ammonium Hydroxide (30% active)	4.0000
EDTA chelating agent	2.0000
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

2. Part A - Product 7

Protocol:

- 1 Add water
- 2 Add ascorbic acid & sodium sulphite
- 3 Add chelant
- 5 Add ammonium hydroxide
- 6 pH adjust with acetic acid to required pH
- 7 Add xanthan gum polymer to propylene glycol
- 8 Mix polymer/glycol with ammoniac/chelant solution
- 7 Add solution to emulsion base slowly and mix thoroughly
- 8 Qs with DI water

Ingredients

a. Product 7

<u>Description</u>	<u>Wt %</u>
Emulsion Base	44.5000
Demineralised water	2.0000
Ascorbic Acid	0.2000
Sodium Sulphite	0.2000
Xanthan Gum (CP Kelco, Keltrol HP)	1.0000
Propylene glycol	6.0000
Ammonium Hydroxide (30% active)	4.0000
EDDS chelating agent (30% active)	26.7000
pH adjust with Acetic Acid to pH 10	qs
qs to 100% with DI water	qs

C. Formulation of Final Peroxide Products

1. Part B - Products 1-10

Protocol:

- 1 Add water
- 2 Add chelants
- 3 Add Hydrogen peroxide
- 4 Add solution to emulsion base slowly and mix thoroughly

Ingredients

<u>Description</u>	<u>Wt %</u>
Emulsion Base	35.0000
Demineralised water	38.6500
DTPA chelating agent	0.2500
HEDP chelating agent	0.1000
Hydrogen Peroxide (35% active)	26.0000

- D. Part A (tint products) and Part B (peroxide products) are mixed in a 1:1 ratio immediately before application to the hair

APPENDIX 2MEASUREMENT OF LIGHTENING AND COLOUR UPTAKE

The instrument used to measure the colour and lightening of the hair was the Hunter LabScan XE spectrophotometer. The settings used were the area view = 12.7 mm and the port size = 17.0 mm. Before each use the instrument was calibrated using a black tile and a white tile. A check for the drift of colour over time was performed each week using a calibrated green tile provide by Hunter.

To measure the colour the hair switch to be measured was placed in an appropriately sized switch holder and made sure that the springs pulled the hair taut and flat against the holder. The hair should be mounted on the white side of the switch holder, to ensure measurement against a white background. The sample was placed face down over the measuring port and the measurement was made. A total of eight readings are taken for all tests; 4 on one side of the switch, and four on the other side, moving the hair switch along its length between readings.

The data generated gives the Lightening level (L). and the change in colour (a and b values). Relevant to this work is the L value, the amount of lightening from the oxidant.

APPENDIX 3MEASUREMENT OF FIBRE DAMAGE USING GONIOPHOTOMETER

Damage caused to the hair was assessed by the Goniophotometer method, which has been established to be suitable for studying the effects of changes in surface condition of the hair (R. F. Stamm, M. L. Garcia and J. J. Fuchs, 'The Optical Properties of Human Hair-I. Fundamental Consideration and Goniophotometer Curves', and 'II. The Lustre of Human Hair Fibres', J Soc. Cosmet. Chem. 28, 571-599 and 601-609 (September 1977)). It has been demonstrated that the shine (gloss or lustre) is proportional to the relative amounts of specularly and diffusely reflected light (I_s and I_d respectively). This is dictated by the refractive index of the fibre and the roughness of the surface. By coating the hair fibres in a very fine coating of gold before measuring the reflected light the internal reflection of the fibre is eliminated and the shine can be used as a sensitive measure of the roughness of the surface. For example, a smooth surface will reflect light that has a large specular content and a small diffuse content.

A GP200 Goniophotometer was used from Murakami Colour Research Laboratory. The gold coating was applied using an Emitech K-500 sputter coater.

Randomly chosen single fibres were loaded onto a single fibre holder (10 fibres per holder) and held in a parallel array. A minimum of 12 holders were loaded giving good reproducibility of $\pm 4\%$. Each single fibre holder was coated in gold using the Emitech sputter coater for 1 minute with a 25mA coating rate. This gives a coating of between 10-300nm of gold on the surface. The sample holder was then loaded into the GP200 Goniophotometer. The following instrumental conditions were employed:

Reflection measurement mode - fixed incident angle, variable receiving angle

Incidence Angle = $+30^\circ$

Detector Angle range = -30° to $+60^\circ$

Light aperture values: Incident = 4.0; Receiving = 2.0

Inclination of specimen table = 0°

Sensitivity = 850

High voltage of photomultiplier = 725

For each set of fibres a reflectance spectrum is obtained. From this spectrum the reflectance peak maximum (I_{max}) is normalised to 1 and all the other reflectances are scaled according to this maximum:

$$I(\text{norm}) = I/I_{max}$$

Where $I(\text{norm})$ = normalised intensity, I = reflectance intensity, I_{max} = reflectance peak maximum.

The shine is calculated from the difference between the specular reflection and diffuse reflection at 0° divided by the width of the specular peak at its half maximum (in angular units)

$$S(\text{norm}) = [(1 - I(0)) / \sigma] * 100$$

Where $S(\text{norm})$ = normalised shine, $I(0)$ = normalised reflectance at 0° , σ = angular full width at half maximum in $^\circ$.

APPENDIX 4**MEASUREMENT OF FIBRE DAMAGE USING SEM GRADING**

At least 40 fibres from each test product were mounted on an SEM stub and prepared for SEM analysis. The middle section of the fibre switch was assessed.

The SEM stub was orientated with the fibres vertical and with cuticles facing downward. The individual fibres were then graded at three different points along the fibre at a magnification of 1000x and at 20KeV. Each fibre was graded according to four classifications:

Low Damage – Cuticle aligned and spaced regularly, some irregularity of the cuticle is allowed.

Mid Damage – Cuticle starts to become very irregularly spaced or some lifting.

High Damage – Cuticle missing, cuticle lifted and some evidence of cuticle lost.

Stripped – Majority of fibre stripped (>90%)

The gradings were recorded on a log sheet and the percentages were calculated of low, medium, high and stripped grades. The percentages of the three grades were then averaged. Finally the Hair Damage Index was calculated as below to give an overall damage number for the hair:

$$\text{Hair Damage Index} = ((1 \times \text{Medium}) + (3 \times \text{High}) + (5 \times \text{Stripped}))/5$$

Note that all graders had been validated for use in technical test methods where the validation involved grading prepared switches to ensure consistent grading.

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